Characterization of SG Iron

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By

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Certificate

This is to certify that the thesis entitled, " **Characterization of SG iron"** submitted by **Vikash Kumar Jha (213MM1468)** in partial fulfillment of the requirements for the award of **Master of Technology Degree** in **Metallurgical and Materials Engineering** at National Institute of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance.

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ABSTRACT

Present investigation is carried out to determine the relation between microstructural aspects and wear properties of spheroidal graphite (SG) cast iron material to be used for production of automotive parts such as gears, camshafts and cylinder pistons. SG cast iron due to its better damping capacity, strength to weight ratio, elongation and tribological properties is the most preferable and widely used material among all type of cast irons. The considerable amount of ductility achieved in SG cast iron is due to the spherical form of graphite rather than flakes as present in grey cast iron which eliminates the stress concentration effect when subjected to different loading conditions and for which it is preferred in automotive parts manufacturing. In order to determine the optimum wear resistance of SG cast iron, the current investigation involves annealing, normalizing, quench & tempering and austempering heat treatments leading to transformation of as-cast matrix to ferritic, pearlitic, tempered martensitic and coarse upper bainitic matrices respectively. Specimens with varying alloying elements were austenitized to 1000°C for 90 minutes, followed by furnace cooling, air cooling, oil and salt bath quenching followed by air cooling for respective heat treatments. Vickers hardness test (at 20Kg applied load) & dry sliding wear test were performed under 20N, 40N & 60N for a distance of 7.54m using Vickers hardness tester & ball-on-plate wear tester respectively. The quench tempered specimen for alloy SG-02 has maximum hardness whereas in case of alloy SG-01 normalized specimen has maximum hardness, the first one has tempered martensitic and second one has pearlitic matrix. The lowest hardness was obtained for annealed specimen having ferritic matrix for both the alloys, whereas the hardness values of other specimen were in between these two for respective alloys. There was not much difference in hardness of ferritic, pearlitic and bainitic matrices, but marginal difference was observed in as-cast and tempered martensitic matrices. The reason for this difference is attributed to the fact that in as-cast condition alloy SG-01(higher hardness) has bull's eye ferritic/pearlitic matrix, whereas that of alloy SG-02 is fully ferritic resulting lower hardness. For quench & tempered as well as austempered specimens alloy SG-01 has lower hardness value than alloy SG-02 because of presence of more C, Mn, Ni, Cr, Mo and Si. These elements may cause strengthening of solid solution of ferrite in ductile cast iron. When operating under 20N load weight gain was observed in every specimen due to the formation of oxides over the worn surface, whereas no such phenomenon was observed for 40N and 60N. The major wear mechanism observed in softer matrix phases were adhesive type signified by delamination layers over graphite nodules while in the harder phase wear rate decreases because of plastic deformation.

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1 Introduction:

1.1 Introduction-

Spheroidal graphite cast iron is a member of cast iron in which graphite is present in the form of nodules. It is also said to be nodular cast iron**.** Due to presence of compact spheroids, it interrupt less with the matrix continuity than graphite flakes. This is the reason that it have higher strength and toughness along with better wear resistance capacity. The carbon percentage present in ductile cast iron and graphite cast iron is same. However spheroidal graphite particle is formed due to addition of some alloying elements such as magnesium and cerium. Magnesium or Cerium helps to increase its dimensions uniformly in all the directions. When silicon is added above 2% it may cause graphitization rate increases. The mechanical properties of SG cast iron can be improved by either addition of alloys or various heat treatment processes [1]. In the grey cast iron, graphite is present in the flaky form due to which stress concentration is more at the sharp corners of these graphite flakes. So it can not able to withstand at high tensile strength and less wear resistive capacity compared to ductile cast iron [2]. This is the reason why ductile cast iron is widely used for industrial application. Due to various heat treatments process different matrix phases is obtained and properties of heat treated sample is also changed with these matrix phases. Metallurgical optical microscope is used to confirm the presence of phases.Austempered and hardened SG iron have preferred characterization than traditional cast iron.

Annealing is a heat treatment process in which ductility of the given sample increases while hardness and strength decreases. This is achieved by means of complete conversion of parent phase into soft ferritic phase. On other hand to increase the hardness and toughness, the sample is heated to austenitizing temperature and then quenched into a salt bath at different rate due to this upper and lower bainite is formed. If the pearlite phase is present in more extent in given sample then the ultimate tensile strength and hardness is increased, while presence of ferritic phase in more extent responsible for ductility [3, 4].

1.2 History of SG iron-

Chinese was first who initially designed cast press in the 5th century BC. In order to make the post and weapons they made a molten metal of iron and discharged it into a previously prepared mould of desired cavity and solidified it to a certain time period by which exact copy of product extracted. Due to its shoddy rate and effectively accessibility it was in wide use in old China. On the other hand, its quality was second rate than steel. Thrown has high compressive strength and it is fragile in character. In light of weak character it less utilized as a part of purposes where a sharp edge is needed. Solid metal is solid under pressure while feeble under strain. Henry VIII introduced the iron cannon in England. Before iron cannons, English were utilizing bronze cannon. After the approach of casting system, cannons being begun to be made of cast iron. Notwithstanding, cannon made of cast iron is heavier than bronze. Thomas Newcomen added to the steam motor which gave a gigantic business of cast iron which was extensively less expensive than metal which was before utilized crude material for making motor chambers.

Keith Millis was developed nodular iron in 1943 which is otherwise called ductile cast iron. Nodular iron is of more fatigue and wear resistance, due to nodular graphite incorporation, as contrast with different members of cast iron.In October 25, 1949, Albert Paul Gangnebin and Norman Boden and Keith Dwight Millis got US patent for nodular iron creation by means of magnesium treatment.

1.3 Chemical composition of SG iron-

In order to improve the properties of SG iron, addition of alloy is done with the specimen. For this purpose different alloying element such as carbon, silicon, manganese, Sulphur and phosphorus is added with the specimen.

1. Carbon-Carbon rate regularly shifts between 3.0 to 4.0 percent. Despite the fact that the most rehearsed carbon rate differs somewhere around 3.4 and 3.8 percent. Cast ability, which is enhanced by enhancing smoothness, is the one of primary parameters which gets impacted by a change in carbon rate.

In SG iron, with the variation in percentage of carbon there is slight change in mechanical properties is observed. With 0.1% of increase of carbon, UTS of ductile iron is lessen by 2.48MPa while very slight reduction in yield strength is observed. Hardness decreases by around 5 numbers every 0.15% expansion of carbon while percentage elongation increases all the more especially in the event of as-cast example. With the different carbon percentage in volume of the given matrix, modulus of rigidity is influenced.

2. Silicon- It is utilized as graphitizer and it expands the spheroids exhibit in the lattice of the SG Iron. It builds the ferrite territory portion by diminishing essential carbides and pearlite. Mechanical properties of SG Iron are extraordinarily affected by the silicon content. For every 0.25% silicon expansion, at 0.18% manganese, there is increment of 21 MPa in UTS of SG Iron by fortifying ferrite network while decreasing rate elongation in annealed ferritized microstructure. Impact resistance is regulated by percentage silicon control and temperature. With the increase in silicon content impact value decreases, whereas on decreasing in temperature impact value increases. Sample of 2.25% silicon gives full ductility in the temperature -10^{0}C. When it is regulated to 1.4%, material possesses ductility even at lower temperature of -60^0C. Manganese & Phosphorous promotes the formation of pearlitic matrix phase but the presence of Silicon overcome the effect of them. So it is noted that the presence of Manganese & Phosphorous should be keep as low as possible [5, 6].

3 Manganese-When manganese is added to the SG iron specimen, it starts the refinement of pearlitic phase together with the stabilization of it. It also decreases the ferritic phase content by the as cast sample. Itobviate the breakdown of pearlite to ferrite by stabilizing the carbide phase and perpetuates the annealing cycle for engenderment of ferritic structures. When its quantity is increased in as cast and annealed specimen, may cause tensile and yield strength is increased. With the increase of Mn content in normalized sample hardenability is increased consequently.

4 Sulphur-Evacuation of sulfur, underneath 0.018%, is critical and vital piece of the creation of SG Iron. Sulfur responds with magnesium and gives magnesium sulphide which captures in the casting, drosses and disables the casting quality. Its content likewise influences the spheroidization of graphite which is advanced by lingering magnesium whose amount is influenced by sulfur. Along these lines, desulphurization is all that much crucial to keep the dross consideration.

5 Phosphorus-when it is added with the given ductile iron sample it creates the brittle phosphide network like with the addition of grey cast iron. It decreases the impact resistance and influence hardness and toughness haphazardly. Phosphorus, especially over 0.08%, raises the weak intense move temperature range. It is subsequently kept lower than 0.043% in the greater part of the castings.

6 Residual Magnesium-Magnesium cumulates firstly with the excess sulphur in the iron sample to compose Mg_2S till it is abstracted to below 0.015%. The magnesium sulphide composed yarely floats to the surface where it can and should be abstracted. Due to high quantity of sulphur in base iron sample, excess quantity of magnesium sulphide slag is formed that may cause casting dross defect is occurred. Due to the fading effect find in magnesium with socking time of the treated molten metal suitable minimum residual magnesium contents is used for heat treatment. In the event that this prompts correspondingly high lingering magnesium substance particularly in the introductory castings beyond any doubt, it can prompt reverse or centerline chill.

1.4 Influence of alloying elements-

(1) Nickel-Nickel in little amount is expected to advance pearlite like manganese yet being a graphitize does not offer ascent to carbides dissimilar to manganese. Nickel substance of 1.0 to 1.5% with suitably regulated manganese may provide carbide free pearlitic as-cast structure.

(2) Molybdenum-Molybdenum is used to increase the pearlitic phase in the given sample of as cast ductile iron. Sometimes it is also used to improve the tensile strength and hardness of materials. Due to addition of 1% of it in the ferritic phase, the increase in tensile strength is observed by 35MPa whereas ductility is reduced by 9%.

(3) Copper-Copper is a type of alloy which is used to mix with magnesium before the start of heat treatment process. It lessens the ferritic phase in nodular cast iron, and provides stability to the pearlitic phase of the same. It enhances the transition temperature along with the reduction of impact resistance.

(4) Chromium- Chromium is added to the sample in order to provide stability to the carbide phase and pealitic phase. When it is added to the austenitic ductile iron then it improves the corrosion resistance together with enhancement of oxidation. To enhance the strength and lessen the growth chromium is mixed with the silicon [7].

1.5 Different microstructures present in nodular cast iron-

(1) Graphite- Carbon is present in nature in two purest form graphite and diamond. Graphite is used as a lubricant due to its soft nature. It is good conductor of heat and electricity but its density is very low. In grey cast iron it is present in the form of flakes while in nodular cast iron it is present in the nodular shape. Nodular shape is responsible for high ductility SG iron.

(2) Carbide-Carbide is present in the form of cementite in cast iron. As we know that cementite is of very hard and brittle in nature. When it is combined with the alloying elements of cast iron like vanadium, chromium and molybdenum it forms carbides. Due to this carbides hardness and wear resistance increases.

(3) Ferrite-As we know that ferrite is of soft in nature. Due to the formation of ferritic phase in the ductile iron sample, improvement in the toughness and ductility is observed. On the other hand strength and hardness is reduced by the formation of ferritic phase.

(4) Pearlite- When the sample having fully austenitic phase of carbon content 0.77% is cooled from austenitizing temperature to eutectoid temperature (723^0C), then austenite phase is transformed into an alternate layer of ferrite and cementite phase called as pearlite phase.

Austenite= ferrite+ cementite (At 723^0C and 0.77% carbon)

Content of pearlite phase improves the strength of sample together with lessen the ductility in required amount.

(5) Bainite-When the sample of fully austenitic phase is cooled from 723^{0}C to below the nose of TTT curve and hold at this temperature for some time then the formation of bainite takes place. Bainite contents ferrite and carbide phase.

(6) Martensite-When the sample is cooled rapidly from a austenitizing temperature to a room temperature then a diffusion less phase is obtained which is called martensitic phase. It is of hard in nature with a residual stress so further tempering is performed over it. Due to tempering toughness increases as well as internal stresses are relieved.

(7) Austenite-Austenite is a type of phase that is obtained in iron carbon diagram. It is a stable phase with FCC structure. In austempered iron, austenite is created by a combination of fast cooling which smothers the arrangement of pearlite and the super saturation of carbon amid austempering, which discourages the begin of the austenite-to martensitic change far underneath room temperature.

1.6 Application of nodular cast iron-

The application of ductile Iron is various and can be discovered in every Industry. While a few of the current applications include substitution of different materials, a stage has been come to in the improvement of the material to legitimacy genuine thoughtfulness regarding configuration segments to suit its own properties to determine full financial and specialized preferences from the utilization of the material.

During the design of product, designer should always focus on the following properties of ductile iron:

1 While designing the complex shapes, it should have excellent fluidity characteristics.

2 It should have good wear resistance property.

3 The corrosion resistance should be more than other type of cast iron.

4 Imperviousness to development and scaling at lifted temperatures, much better than that of grey cast irons.

Due to such high multifarious properties, it is used for various industrial application in comparison of other member of cast iron family. Manufacturing of automobile parts and hollow pipes are the main area of application of ductile cast iron because of its good wear resistance properties [8-10].

Fig 1.6.1: Automobile parts **Fig 1.6.2: Manufacturing of hollow pipes**

It is used in the manufacturing of hollow pipe by several pipe manufacturing companies. It is used in order to store and transfer the high pressurized fluid. To withstand such a high elevated temperature and pressure the material of pipe should be made up of ductile cast iron that provides it high strength in addition with high toughness.

It is also used in the production of crankshaft of S.I. and C.I. engines. During the working of engine crankshaft has to be withstand at variable load conditions with a sudden jerk so it should be of high strength in addition with superior toughness. Ductile cast iron fulfills all such type of requirements.

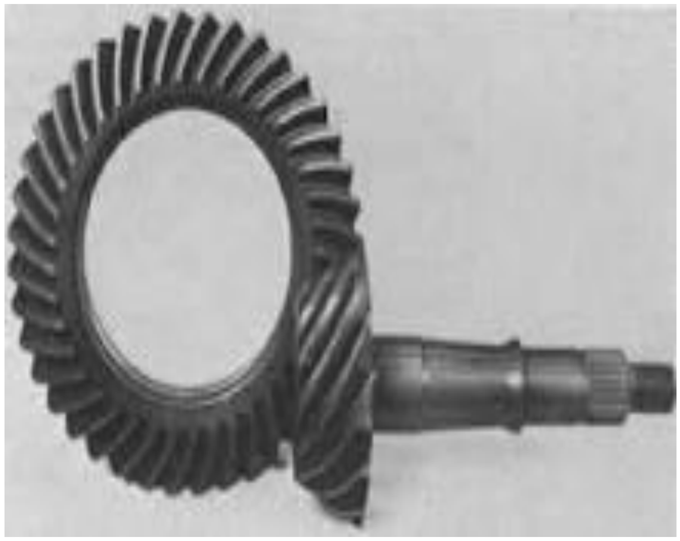


Fig 1.6.3: gear manufacturing

Spheroidal graphite iron is used in the manufacturing of gears because of its high toughness together with strength. For gear designing it is necessary that it can withstand at high enough load without any breakage.

Chapter

2

Literature Review

2 Literature review:

Mahmoud Hafiz [11] suggests that when austempering is performed over the sample of ductile cast iron then improvement in mechanical properties is observed. To do experiment, different ductile iron samples is subjected to isothermal austempering temperature which is variable for different samples. The sample is first heated to an austenitizing temperature 1183K and then quenching is done at different rate of 593K and 723K, respectively. The sample that has cooled to a temperature 593K, again heated to a temperature 723K, whereas latter has cooled consequently to 593K. As a result of the experiment it was observe that yield strength of first sample was 2% more than second sample while ductility of second sample was observe three times more than first sample.

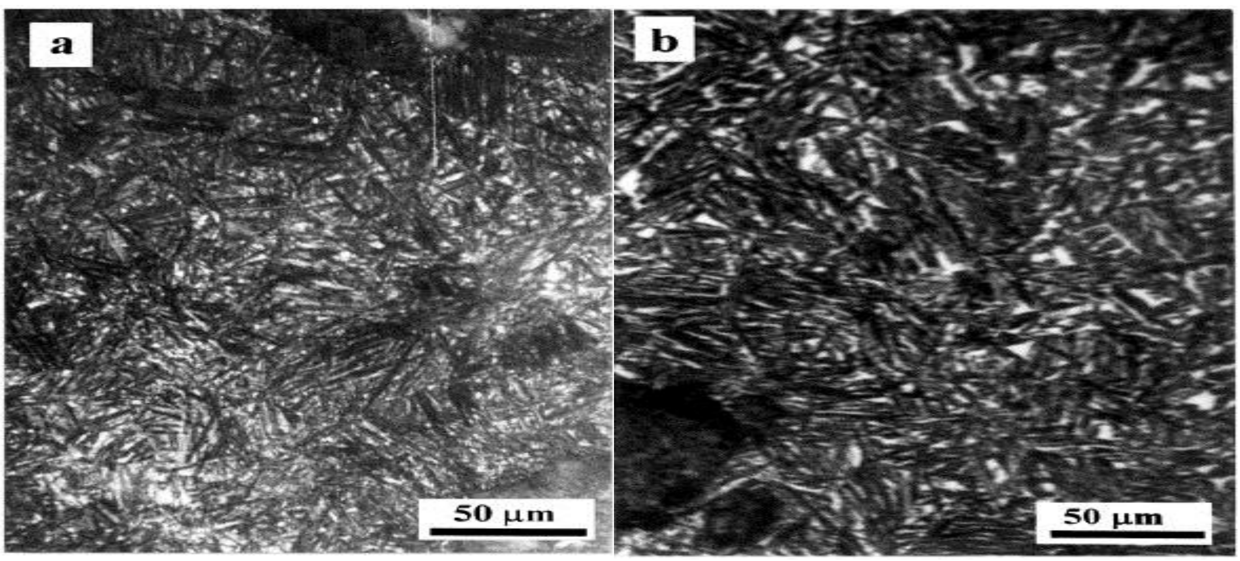
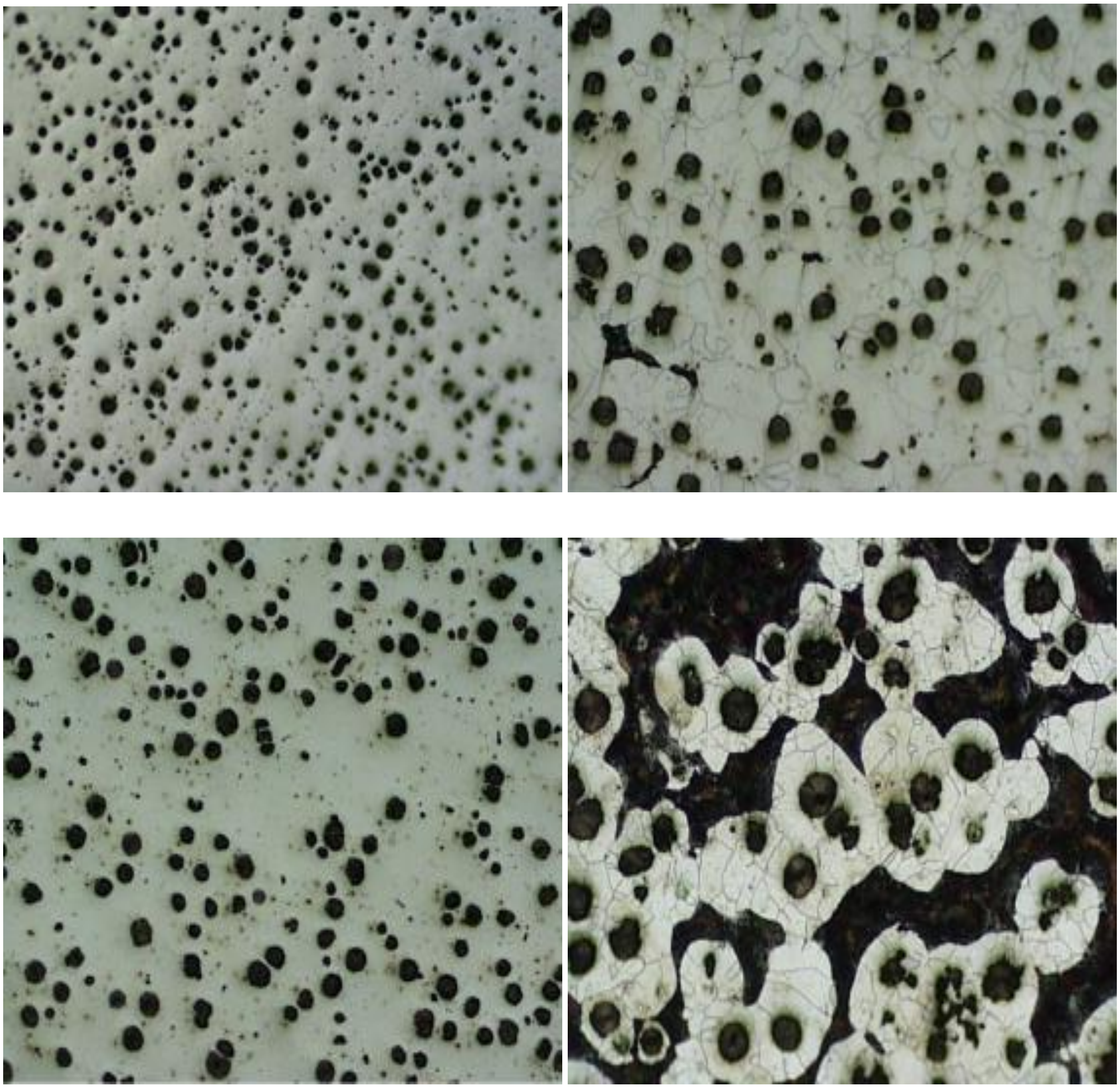


Fig 2.1: Microstructures of sample heated to austenitizing temperature of 1183K for 3.6ks then austempered at constant temperature of (a) 593K and (b) 723K, for 5.4ks.(Reference[11])

Sheng Da and Zheng Yongping [12] propose that cast iron pipe can be manufactured by three basic casting processes-(a) continuous castings, (b) centrifugal castings and (c) manual castings. Centrifugal casting techniques is generally not used because of too much investment so continuous casting is generally preferred over the centrifugal casting. Continuous casting is performed by mixing REMgSiFe. According to author, when such elements are mixed with ductile iron pipe or heat treatment is performed over it, may cause both structure and characteristics are changed. Before the heat treatment, the strength of it was observe to be 515-485MPa, whereas elongation was observed to be 2.5-2.7%. After the heat treatment, strength and elongation were observe to be changed to 440-425MPa and 7-16% respectively. Microstructures of ductile cast iron were observe to have ferritic phase, pearlitic phase and both ferritic and pearlitic phase.

Fordyce E. P. and Allen C. [13] have suggested that wear response of austempered ductile cast under the dry sliding conditions. Pin on Disc type wear test is performed over the sample. As a result of the test wear rate of austempered sample is find to be less than basic as cast sample. During the experiment sliding velocity is varied in between the range 0.5m/s-2.0m/s under the load of 2MPa. AS a result of whole experiment it is observed that at lower speed there is high wear rate while at higher speed mild wear rate. This difference is found because of oxide formation and surface hardening characteristics.

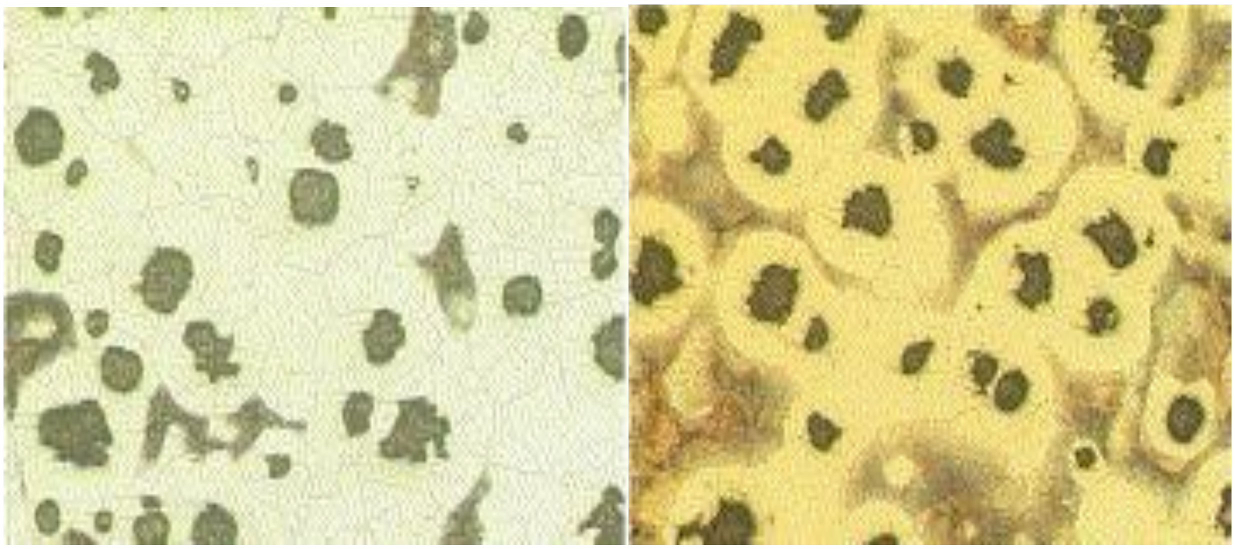
Gonzaga R.A.[14] says that mechanical properties of SG iron changes due to the formation ferrite and pearlite phase. When the pearlitic content in ductile cast iron is more, yield and tensileStrength of it increases. Sometimes it is also observe that due to graphite particle shapes and second phase formation tensile strength and yield strength are changed. Above figures shows that pearlitic and ferritic both phases are present in SG iron. Ferritic phase improves ductility while pearlitic phase may cause hardness increases. Images are found to be clear by the use of etchant (natal 2%).



(a) before etching **(b) after etching**

Fig 2.2: Microstructures of different matrix phases (100X)-(reference [14])

Konoplyuk S.[15] says that the characteristic and properties of nodular cast iron can be determined by means of a method of eddy current. For this purpose FCD 450-600 grades sample of cast iron is used. After the supply of eddy current response tensile strength and hardness is correlated. On the other hand ultrasonic test is not providing a good response in comparison of eddy current test.



(a) Microstructure with ferritic phase **(b) Microstructure with pearlitic phase**

Fig 2.3 Different microstructures are obtained- with ferrite & pearlite matrix phase (reference [15])

Cardoso P.H.S. et al. [16] introduced a work to calculate the different mechanical properties as well as the wear properties exhibited by a family of ductile iron and white cast iron. For this purpose he prepared samples and then heat treatment was performed over them. Different properties of samples was determine with the help of optical microscopy and SEM techniques. Wear test was done on a rotating device. As a result of this mass loss was observe. It was found that more weight loss occur in the white cast iron in comparison of ductile cast iron until 120h of testing ,whereas between the range 144h- 196h result was reverse. This was observe because of presence of retained austenite in nodular cast iron. On the other hand if there was rise in toughness during the impact test may cause resistance to abrasion was also increased due to transformation of retained austenite into the martensite.

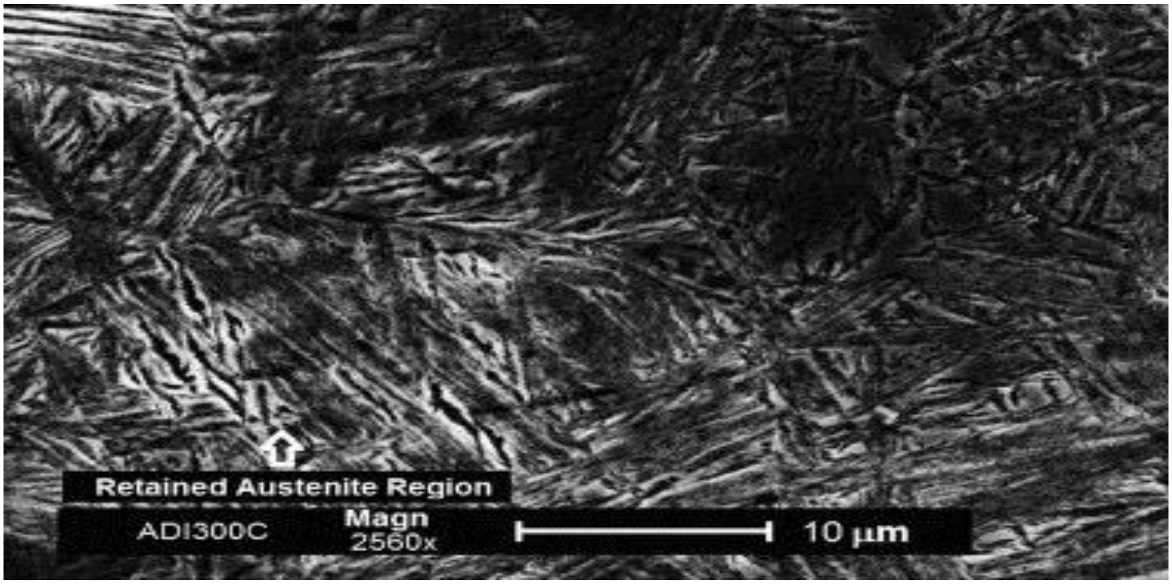


Fig 2.4: Retained austenite transform into martensite (reference [16])

Yoon-Jun Kim and et al. [17] have given information about the different mechanical properties of austempered ductile cast iron. Sample was first mixed with copper and molybdenum, then heated to a temperature of 910^0C for time period of 90min. After that sample was cooled into a salt bath maintaining the temperature 350^0C – 410^0C. Due to this treatment there was improvement in the different mechanical properties was observe such as tensile strength, ductility and fracture toughness. On the basis of the following investigation, it was conclude that austempered ductile cast iron that was formed at higher temperature (between 390^0C- 410^0C) was ASTM grade 1, whereas other was of ASTM grade 2.

Hernandez J.L. and et al. [18] had got idea to investigate the microstructures and mechanical properties of alloyed ductile cast iron, when it was experience a conventional austempering and stepped austempering processes in addition with XRD techniques. XRD techniques was done in order to get information about phase transformation during the conventional austempering and stepped austempering processes. When former process was done some site of sample remained untransformed whereas later may cause these sites disappeared and at these site fine austenite was formed. This concluded that stepped austempering had more advantage over conventional austempering process.

Abedi H. R. [19] while working for wear determination of SG iron samples at different load conditions he found that the ductile cast iron having ferrite and pearlite phase was influenced due to presence of graphite nodule count under the dry sliding condition. For the purpose of doing work he took several samples of nodular iron of different thickness and different nodule count. Heat treatment was done prior to the wear in order to arrange the matrices of ferritic and pearlitic phase. Wear test was conducted on pin on disc type apparatus under the condition of dry slide. As a result of this he concluded that at the lower load oxidation wear was occur while at higher load condition adhesive wear involved for wear mechanism. Also those sample which had higher count of nodule exhibited lower wear rate, whereas at higher load wear resistance increased with the increasing nodule count.

Rao P. Prasad and PutatundaSusill K. [20] investigated that fracture toughness of austempered ductile cast iron was depend on the microstructures. In order to obtain the microstructure it was mixed with nickel, copper and molybdenum and then heated to austenitizing temperature. The characterization of microstructure was done with the help of optical microscopy and XRD techniques. Finally he got that the microstructure due to the formation of lower bainite had more fracture toughness in comparison of upper bainite microstructure. Due to presence of volume fraction of retained austenite and carbon fracture toughness changed. If the volume fraction of retained austenite was 25% then maximum fracture toughness was observed.

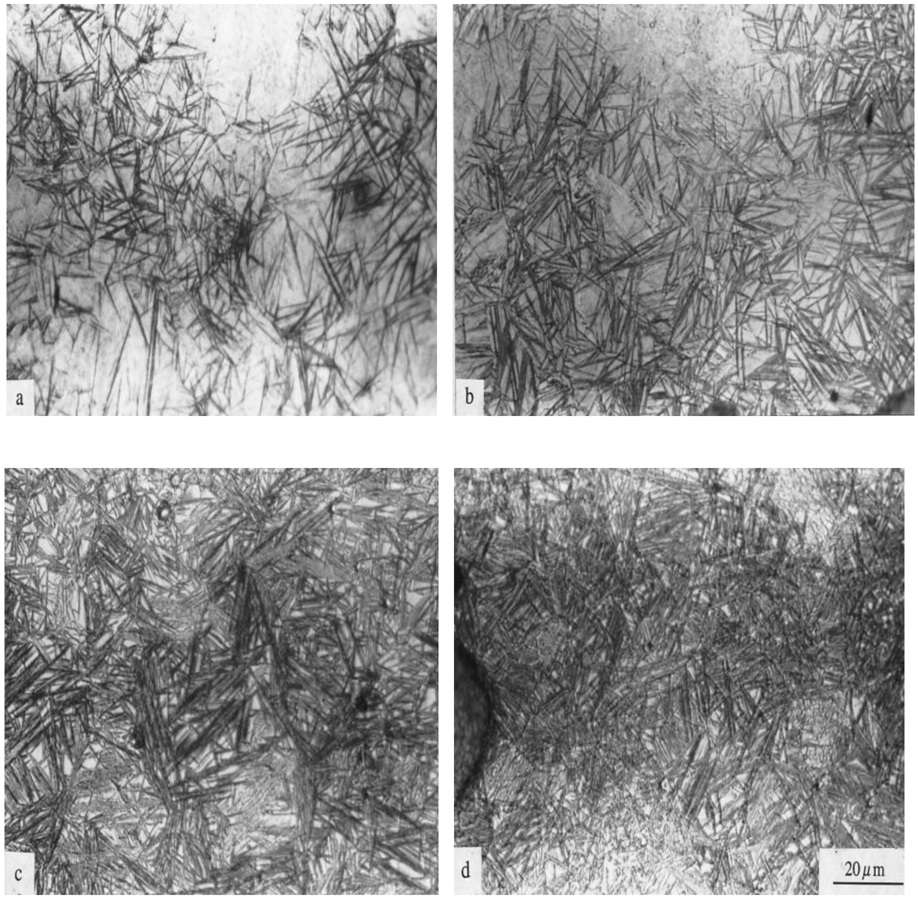


Fig 2.5: Different microstructures obtained by austempering heat treatment at 288°C for different times (reference [20])

Islam M. A. [21] proposes that wear response of as-cast sample and heat treated sample is different when they are subjected to dry sliding condition. Wear measurement is done by means of pin on disc type apparatus. Both as-cast and heat treated sample are subjected to 1.5Kg load under a sliding speed 0.88m/s. Whole experiment has done under atmospheric conditions. Wear rate can be measured by means of weight loss of samples and optical microscopy techniques. Wear measurement is done after 9500m of sliding distance and observe to be wear rate of as cast sample is three times more than heat treated sample. Wear rate in as-cast sample is due to adhesive wear, whereas abrasive wear is responsible of wear in heat treated sample.

Chapter

3

Experimental procedures

- Sample Preparation
- Heat treatment
- Characterization
  - ✓ Optical microscopy & XRD Techniques
- Vicker's Hardness test & Wear measurement
- FESEM

3 Experimental details:

3.1 Sample preparation-

The main goal of this project is to investigate different microstructures obtained after the heat treatment and correlates microstructures with the wear properties of ductile cast iron. The specimen of SG-01 & SG-02 composition is shown below in the table 1. The given samples were subjected to different machining processes prior to heat treatment and after the heat treatment the oxide layer is removed by means of filling operation.

Table 1: chemical composition of ductile iron in wt. %.

Alloy	Elements (in wt. %)											
	C	Si	Mn	S	P	Cr	Ni	Cu	Mo	Mg	Ce	Fe
SG-01	3.45	2.07	0.15	0.008	0.024	0.02	0.15	----	----	0.043	----	Rest
SG-02	3.61	2.10	0.20	0.007	0.022	0.03	0.47	0.009	0.001	0.043	0.004	Rest

3.2 Heat Treatment-

Heat treatment was done on muffle furnace. In order to get required microstructure different heat treatment processes was performed over the sample such as Annealing, Normalizing, Tempering and austempering.

3.2.1 Annealing- Annealing is conducted on SG iron in order to provide good machinability and to improve ductility. The given sample was first heated to an austenitizing temperature of 1000^0C and holding at this temperature for 90min. then furnace cooling was done to a temperature of 700^0C, maintaining at this temperature for 5hr 30min then followed by furnace cooling to a room temperature. As a result of annealing generally ferritic matrix phase was formed in which spheroidal graphite logged. As we know that ferritic phase is softer in nature so it may cause ductility increases.

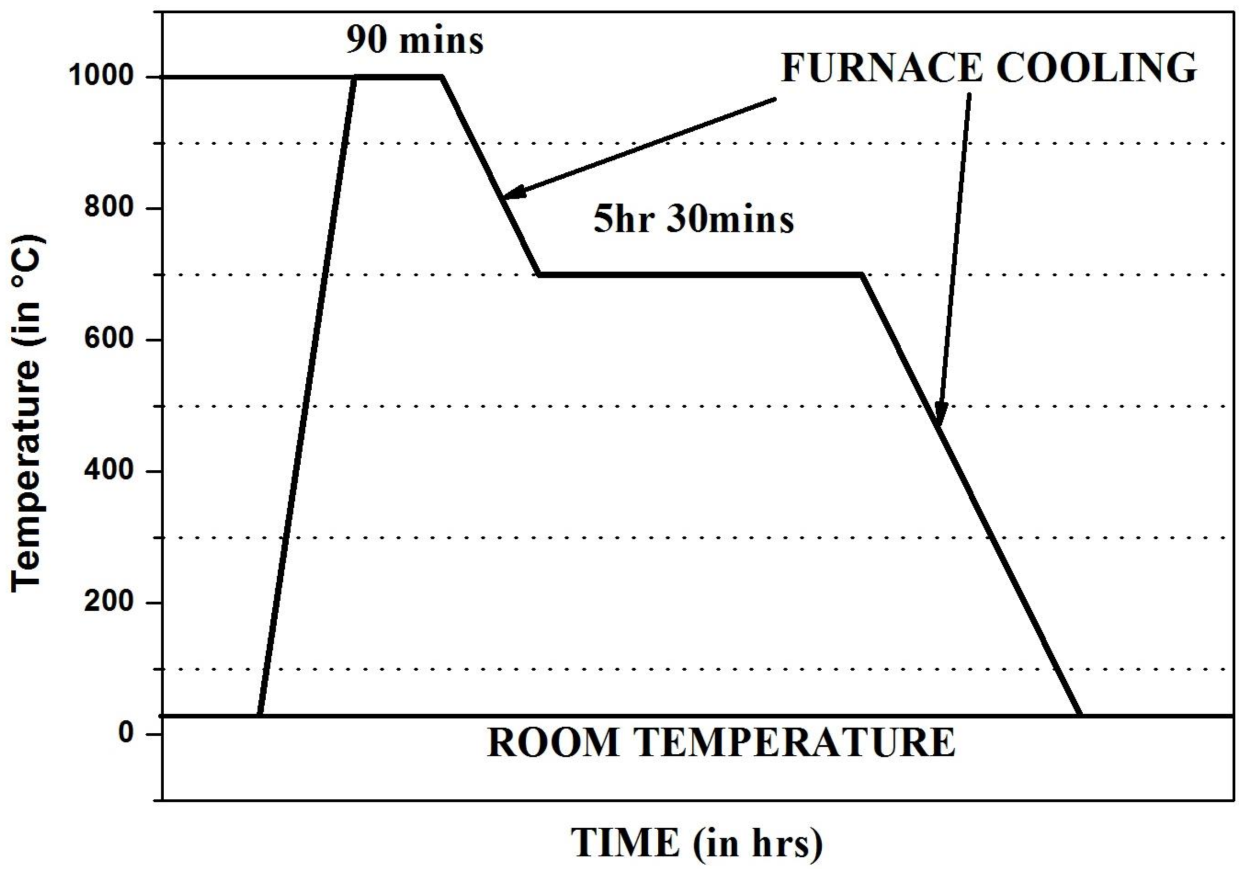


Fig 3.2.1: Diagram shows Annealing heat treatment

3.2.2 Normalizing- Normalizing is conducted in order to improve hardness and strength. Given sample was first heated to an austenitizing temperature 1000^0C and holding it for 90min. then followed by air cooling to a room temperature. Due to normalizing pearlite matrix phase was obtained. Pearlitic phase may cause resistance to mechanical properties increases. When alloying element such as nickel, molybdenum and manganese is added to the sample then tendency of formation of pearlitic phase increases.

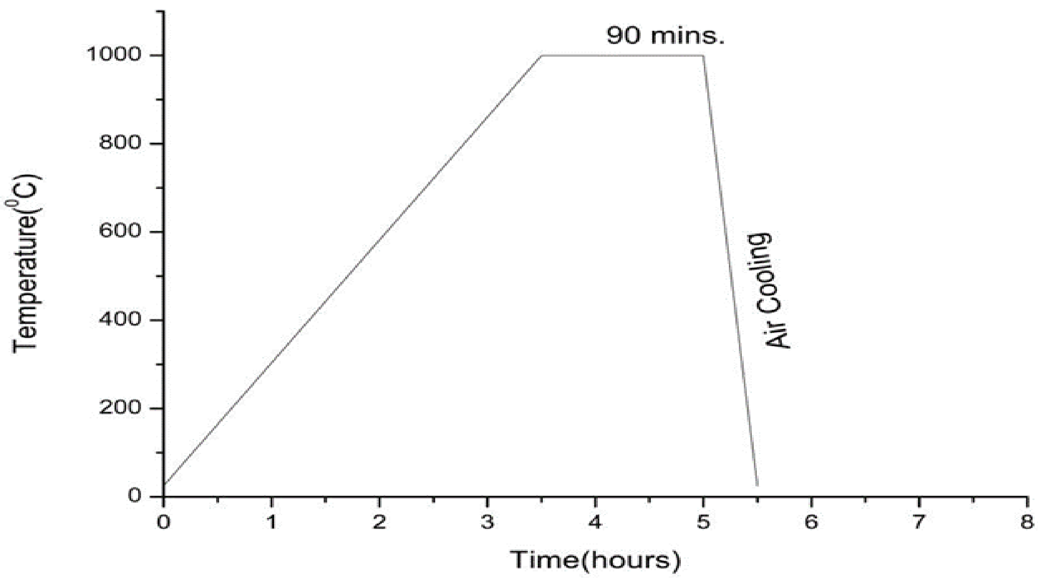


Fig 3.2.2: Diagram shows Normalizing heat treatment

3.2.3 Tempering- When the cooling is done at a very fast rate then diffusion less phase is formed called martensitic phase. This is very hard and brittle in nature and having internal stresses. In order to relive internal stresses and increase the toughness tempering was conducted on the sample. Given sample was first heated to an austenitizing temperature 1000^0C, holding it at that temperature for soaking time 90min. then oil quenched at 100^0C for tempering time 2 hours followed by air cooling to room temperature. After tempering heat treatment, quenched tempered martensitic matrix phase is obtained. Figure of quenched & tempered heat treatment is shown below-

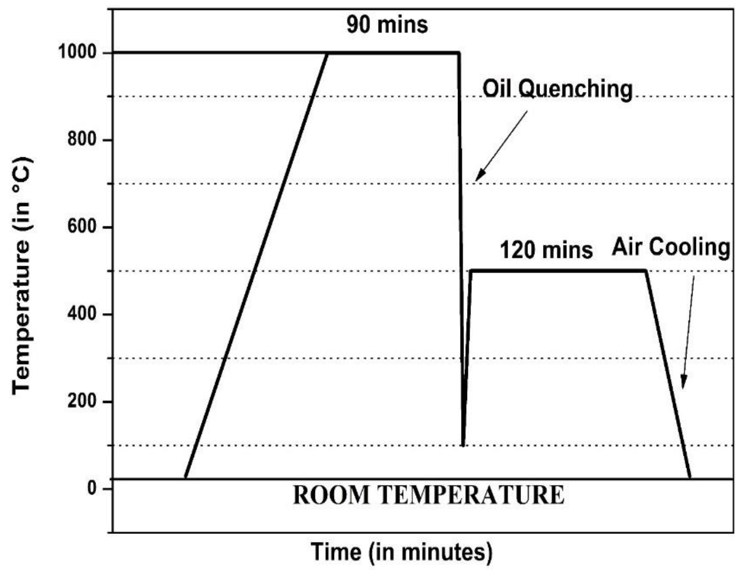


Fig 3.2.3 Hardening and tempering Heat treatment

3.2.4 Austempering- Given sample was first heated to an austenitizing temperature 1000^0C, holding it at this temperature for holding time 90min. then cooled into a salt bath maintaining the temperature of 500^0C, holding at this temperature for 240min. then followed by air cooling to a room temperature. Due to austempering heat treatment, upper coarse bainite matrix was formed.

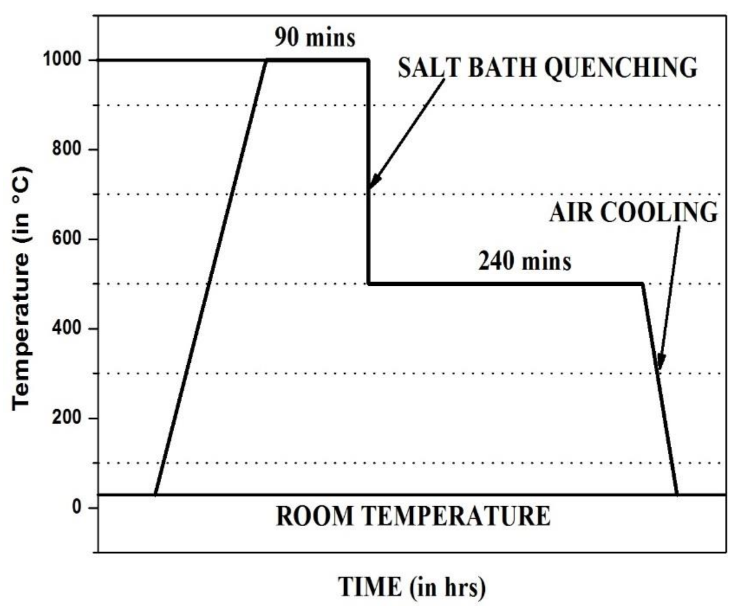


Fig 3.2.4: Diagram of Austempering heat treatment

3.3 Characterization-

3.3.1 Optical microscopy and XRD techniques-

Optical microscope and image analyzer are used to determine the microstructures, nodularity, nodule counts/mm^2 of as-cast and heat treated specimen of SG-01 and SG-02 composition.

Fig 3.3.1: Image of optical microscope

X-ray diffraction Technique is used to determine the different matrix phases present in as-cast and heat treated specimen of the composition SG-01 and SG-02 respectively. XRD was carried out at voltage of 30KV with the help of Cu target diffractometer. Scanning were performed over different specimen between the angular ranges of 40^o-90^o with a constant speed. The different profile peaks were undergo for analysis on computer and intensities were observed.

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Fig 3.3.2: Image of XRD

3.4 Vicker's Hardness test and wear measurement-

Hardness is the property of material by virtue of which it can resist indentation and scratches. Vicker's hardness test is conducted over the samples in order to determine the hardness. Hardness can be measured on the basis of square- based diamond indenter. It has an angle of 136^0 between the opposite faces. Hardness is decided on the basis of vicker's pyramid number (VPN).

Hardness was measured by the Vickers hardness tester for dwell time 10 s under the application of load of 20 kg.

Fig 3.4.1: Vicker's Hardness Tester machine

Wear test was conducted over the samples on DUCOM TR-208-M1 Ball on plate type wear monitor under the load of 40N, 50N and 60N respectively under dry sliding condition. The sliding distance was 7.54m and linear sliding velocity was 0.063m/s. Weight loss was measured by means of electronic balance of 0.1mg accuracy. Before the weight measurement samples were cleaned with acetone.

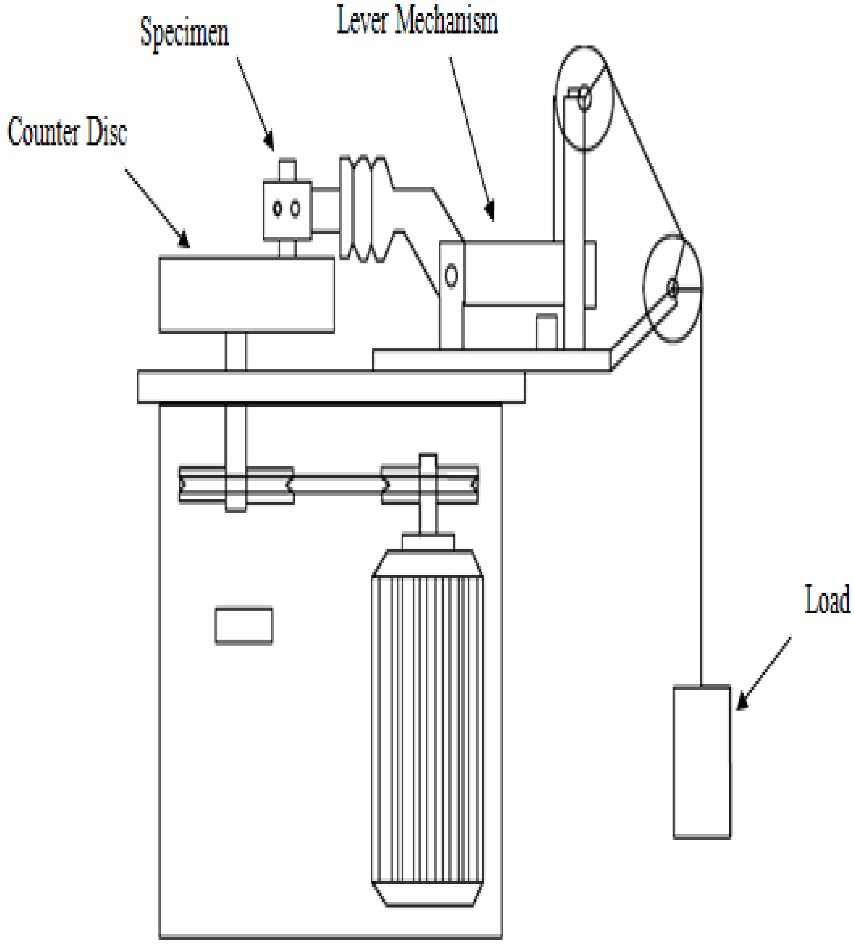


Fig 3.4.2: Mechanism involved in Ball on plate type tester

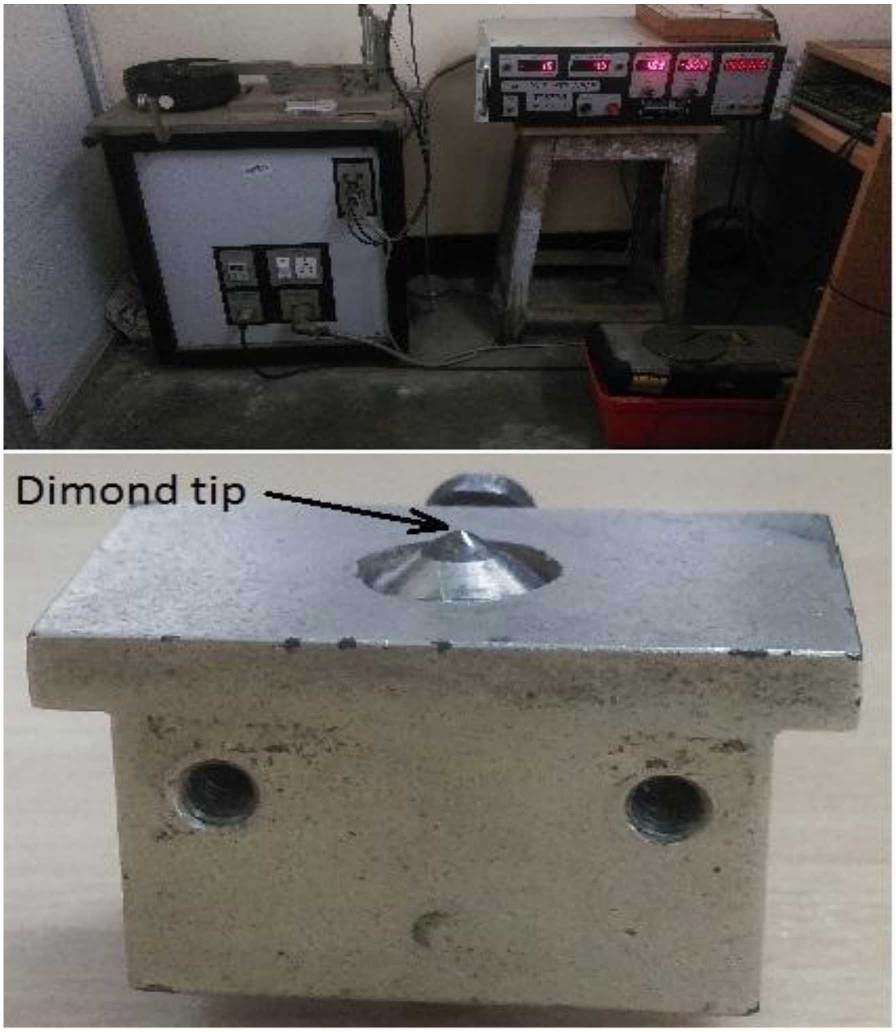


Fig 3.4.3 DUCOM TR-208-M1 Ball on plate type wear monitor

3.5 FESEM-

The full name of FESEM is field emission scanning electron microscope. As the name implies electron is used in order to scan the surface. A field emission source is always required to emit electrons. In order to find out the contaminated spot along with the worn surfaces micro FESEM or EDAX is used. Images of FESEM is clearer than that of SEM.

Chapter

4

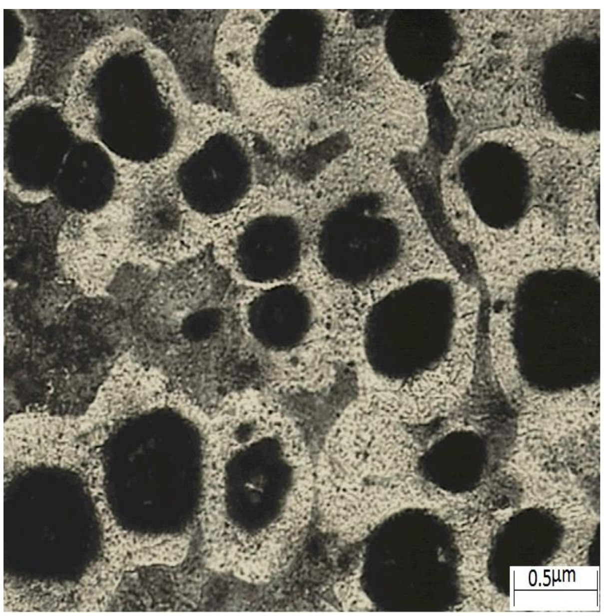
Results and Discussion

- *Microstructural investigation*
- *XRD analysis*
- *Hardness and wear measurements*
- *Wear mechanism*

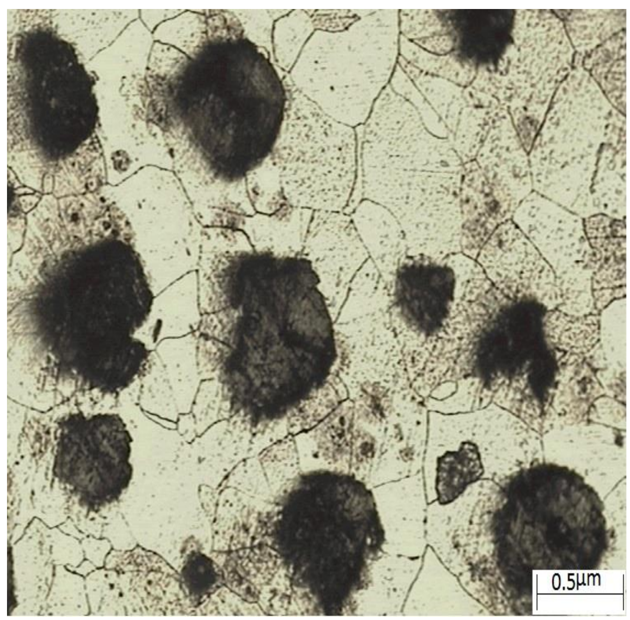
4 Results and Discussions:

4.1 Microstructural Investigation-

Metallographic technique is used to investigate and correlate the wear system response with different matrix microstructures. Prior to investigation different samples of SG iron were polished with the help of belt polisher and then made to pass through several grades of emery paper followed by cloth polishing and diamond polishing. Finally surface of specimen were cleaned in natal solution. After that microstructures were seen by means of optical microscope with image magnification 100X. Different images of microstructures of as cast sample as well as heat treated samples are shown below-

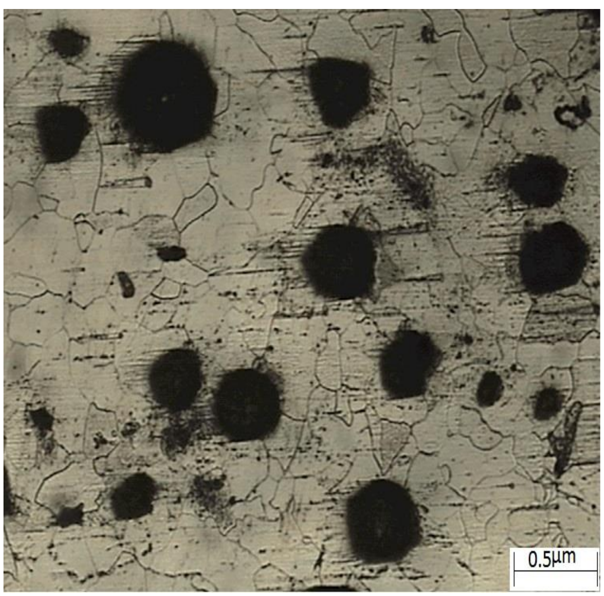


(a)As- cast SG-01

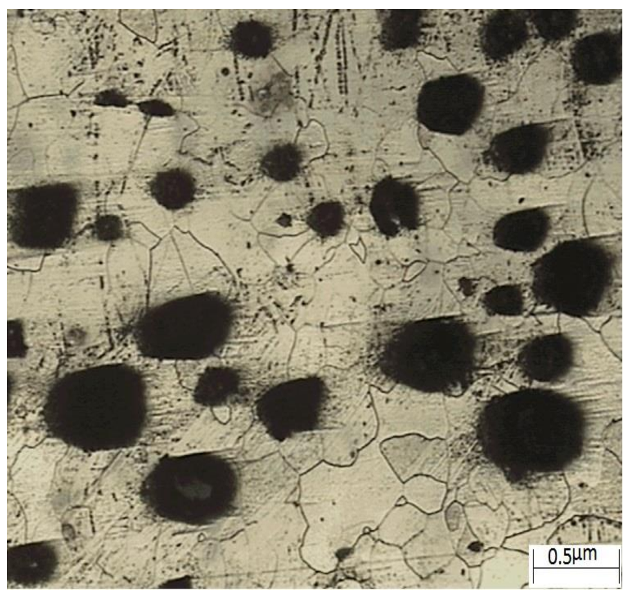


(b) As-cast SG-02

From the figure (a) it is clear that as cast sample of SG-01 composition has bull's eye ferritic/ pearlitic structure, graphite (black) nodules are surrounded by white ferritic phase along with greyish pearlitic phase, whereas from the figure (b) it is clear that as-cast sample of SG-02 composition has fully ferritic matrix in which graphite nodules (black) are embedded. The reason behind the fully ferritic matrix in SG-02 is presence of higher amount of Si which has tendency to promote the ferritic phase. Si increases dissolution of carbon in matrix phase. Due to this high dissolution of carbon there is more tendency to formation of ferritic phase in SG-02 composition (ferrite = 71% and graphite content =29%) in comparison of SG-01 composition. Copper and molybdenum is also present in SG-02 composition but in lower extent, which are the pearlitic stabilizers but their effect is overcome by the presence of Si content. Therefore they are not able to promote pearlite phase.



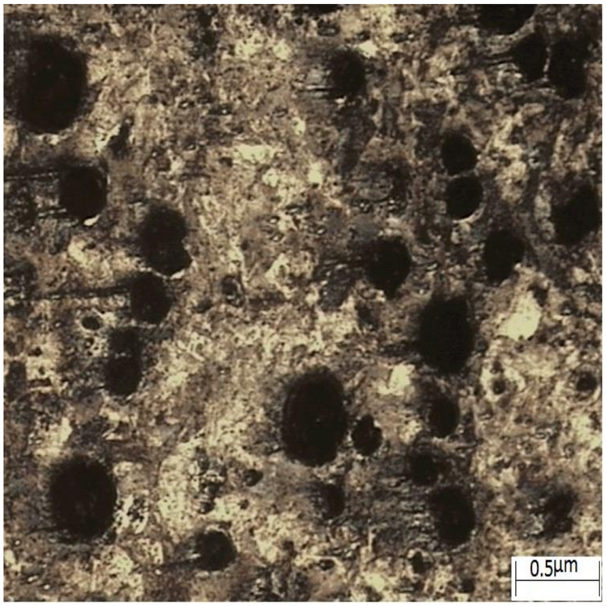
(c)Annealed SG-01

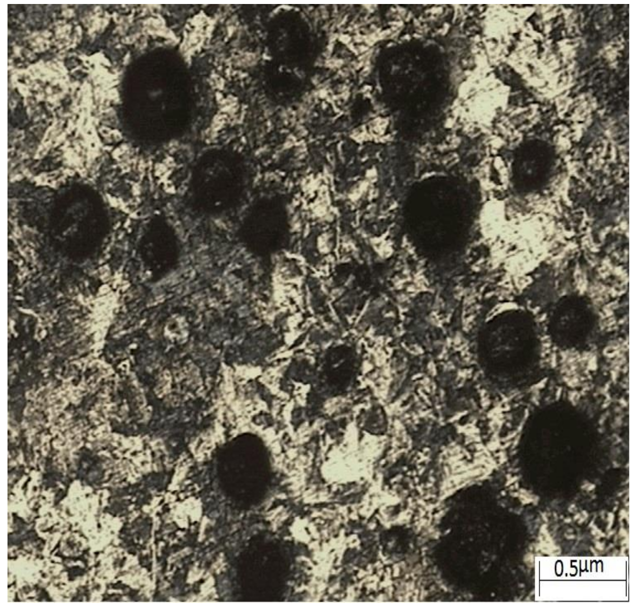


(d) Annealed SG-02

From the above it has been observe that there is not so much difference in the microstructures of annealed sample of SG-01 and SG-02. Figure (c) and figure (d) both have graphite nodule (black) is present in the fully ferritic matrix phase.

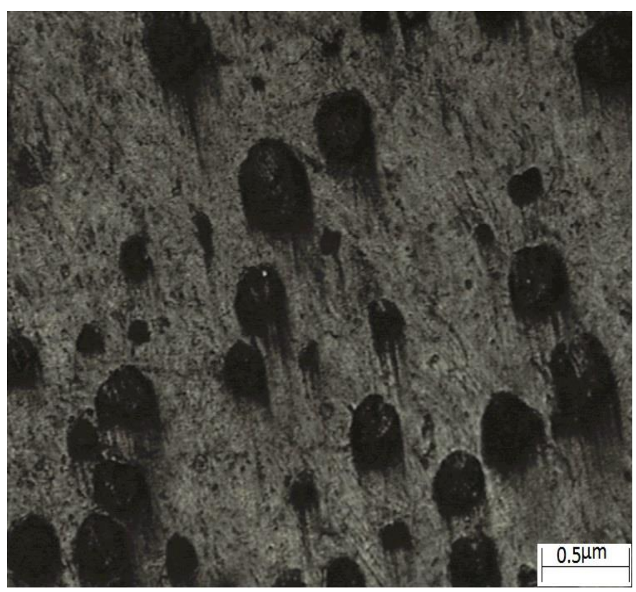
From the figure (e) it has been observe that normalized sample of SG-01 composition and SG-02 composition have ferritic (white)/pearlitic (grey) phase in which graphite nodules (black) are embedded.





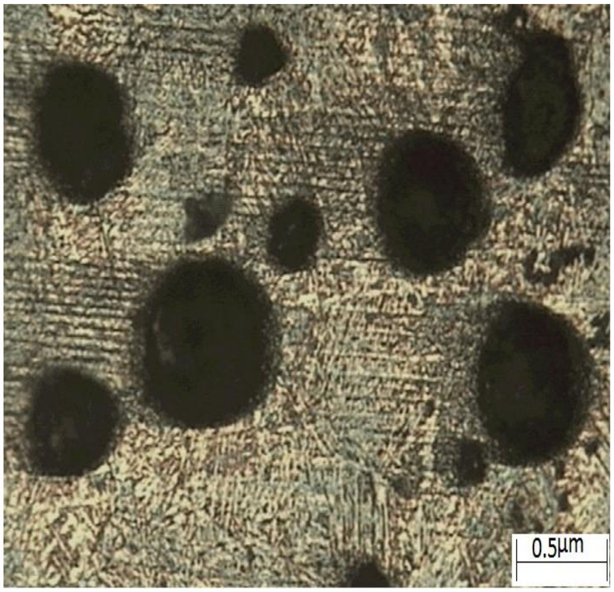
(e) Normalized SG-01 **(f) Normalized SG-02**

Mo, Cu and Ni are the elements that increase the pearlitic phase but their effect is reduced by means of higher content of Silicon. After the analysis through metallography technique it has revealed that SG-02 composition has less than 29% pearlite phase in comparison of SG-01 composition.

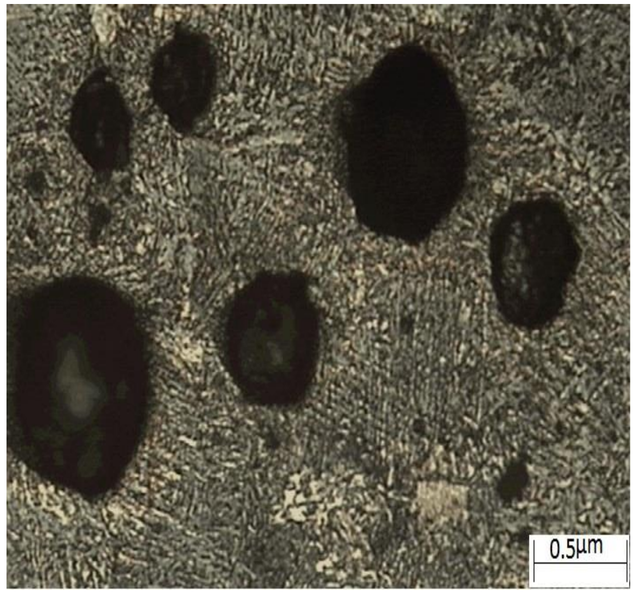


(g) Tempered SG-01 **(h) Tempered SG-02**

Above figures reveals the fact that for the different composition of sample same microstructure are obtained in the case of quenched and tempered. It has number of graphite nodules (black) with tempered martensitic matrix phase.



(i) Austempered SG-01



(j) Austempered SG-02

Fig 4.1: represents different microstructures obtained by various heat treatments

From the above figure it has been revealed that when austempering heat treatment is done over the samples of different composition then a number of graphite nodules are embedded in the ausferritic matrix phase.

Table 2 and 3 show that how does the nodule counts, nodularity and area of different matrix phases change in as- cast and heat treated specimen of SG-01 & SG-02 composition. From the table it is clear that nodule count/$mm^2$is a function of cooling rate. When increasing cooling rates are employed in various heat treatment process then nodule counts increases consequently. Nodule counts is found to be maximum for austempered specimen and lowest for as- cast specimen for SG-01 composition. But in case of SG-02 composition highest nodule count is obtained for quenched & tempered specimen while lowest for annealed specimen. When the cooling rate is so fast, nodule counts increases, whereas in case of slower cooling rate nodule counts decreases. This is attributed to the fact that due to faster cooling rate, the diffusion of carbon atoms are restricted so they are not able to migrate from one site to another site and start to nucleate at their original sites, whereas no such things is observed in case of slower cooling rate. On the other hand nodularity is almost inversely proportion to nodule counts/mm^2. It can be defined as degree of roundness of any species. Where, F= ferrite, P=Pearlite, G=Graphite, M=tempered martensite and B=Bainite. Table 2 and 3 shows nodularity, nodule counts/mm^2 and area fractions of different matrix phases along with graphite area fraction-

Table 2 and Table 3-

Alloy			SG-01				
			As-cast	Annealed	Normalized	Quenched & tempered	Austempered
Microstructural Entities	Nodularity		96%	99%	97%	89.5%	94%
	Nodular count (mm^2)		19	23	37	48	57
	Area Fraction	F	48%	80%	19.5%	2.5%	--------
		P	13.6%	--------	65.5%	---------	---------
		G	38.4%	20%	15%	40%	23%
		M	------	-------	--------	57.5%	--------
		B	-------	-------	-------	--------	77%

Alloy			SG-02				
			As-cast	Annealed	Normalized	Quenched & tempered	Austempered
Microstructural Entities	Nodularity		94%	95.5%	95%	98.5%	90%
	Nodular count (mm^2)		33	25	35	65	58
	Area Fraction	F	71%	75%	21.5%	--------	--------
		P	-------	--------	46.5%	---------	---------
		G	29%	25%	32%	24%	24.5%
		M	------	-------	--------	76%	--------
		B	-------	-------	-------	--------	75.5%

4.2 XRD analysis-

The combined patterns are obtained when x ray diffraction analysis of SG-01&SG-02 composition is done for 2θ range 40^{o}-90^{o}. On the basis of pattern we can see that peaks are formed for crystallography planes (110), (200) and (211) represents BCC structure. This BCC structure indicates that phases are either ferritic, pearlitic or martensitic matrix phase. The conformation of matrix phase is accomplished by microscopic analysis. For the 2θ value of about 64.1 we got crystallographic plane (200) along with the plane (311) it explains the presence of ausferritic phase.

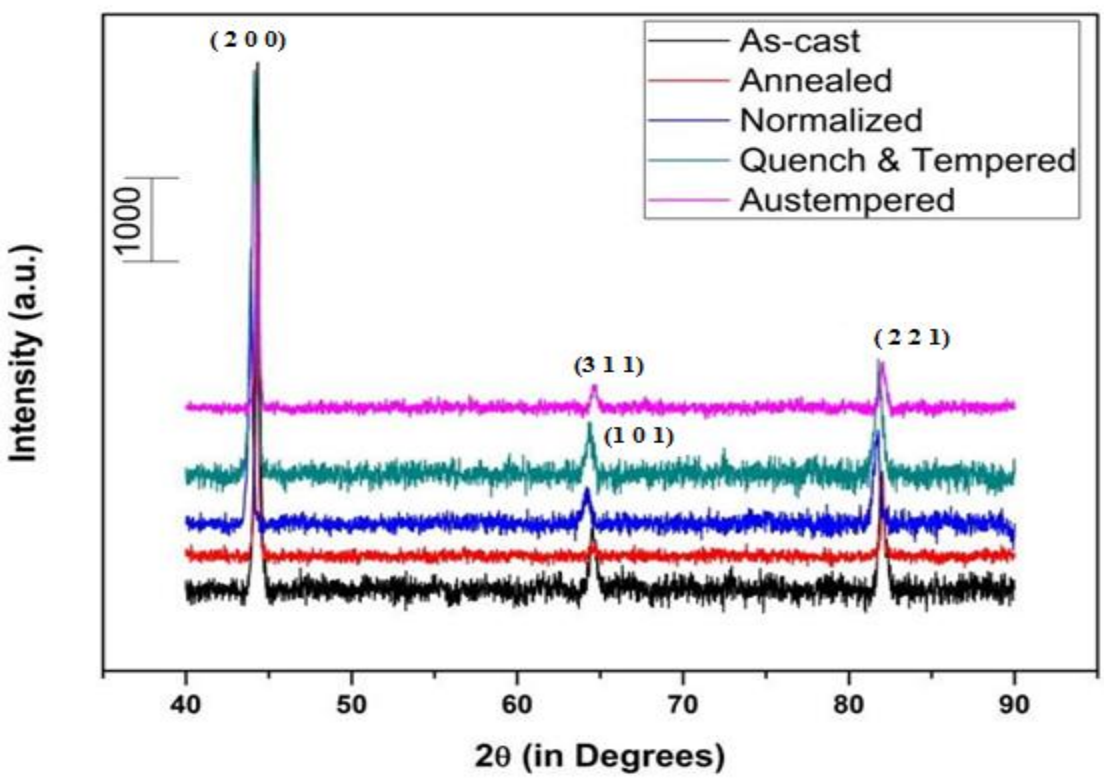


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Fig 4.2: Peaks of XRD analysis

4.3 Hardness and Wear properties-

Hardness test was conducted on the Vickers Hardness tester for both SG-01&SG-02 composition. The following graph is plotted in order to compare the hardness of both SG-01&SG-02 on the basis of data obtained for from the test. From the plot it is clear that hardness of as-cast specimen of SG-01 composition (red line) is more than that of as-cast of SG-02 (black line). This is due to the fact that presence of bull's eye structure of ferrite/pearlite matrix phase in former case and presence of fully ferritic matrix phase in later case. On the other hand in case of annealed specimen there is not so much difference in the hardness because of presence of ferritic phase. Only slight difference is due to the fact of presence of Si content in SG-02 composition is more than that of SG-01 composition which promotes the formation of ferritic phase. So presence of ferritic (softer) is more in annealed specimen of SG-02.

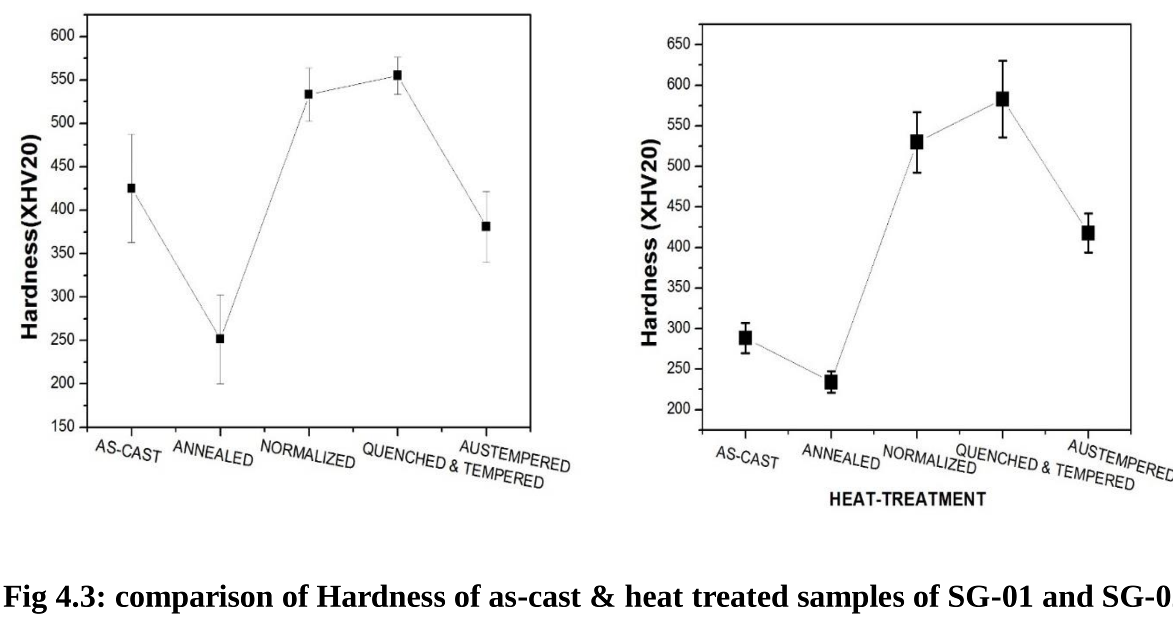


Fig 4.3: comparison of Hardness of as-cast & heat treated samples of SG-01 and SG-02

Furthermore, in case of normalization, the hardness of normalized specimen of SG-01 is more in comparison of SG-02. This is because of Mo, Cu and Ni elements that increase the pearlitic phase but their effect is reduced by means of higher content of Silicon in SG-02 composition. After the analysis through metallography technique it is revealed that SG-02 composition has less than 29% pearlite phase in comparison of SG-01 composition. For quench & tempered as well as austempered specimens alloy SG-01 has lower hardness value than alloy SG-02 because of presence of more C, Mn, Ni, Cr, Mo and Si in later one. These elements may cause strengthening of solid solution of ferrite in ductile cast iron. The quench tempered specimen for both the alloys have maximum hardness value.

Wear test was conducted on DUCOM TR-208 M1 ball on plate monitor under 20N, 40N and 60N load respectively for the sliding distance 7.54m with a sliding speed 0.063m/s. A plot between the weight loss and load is drawn below-

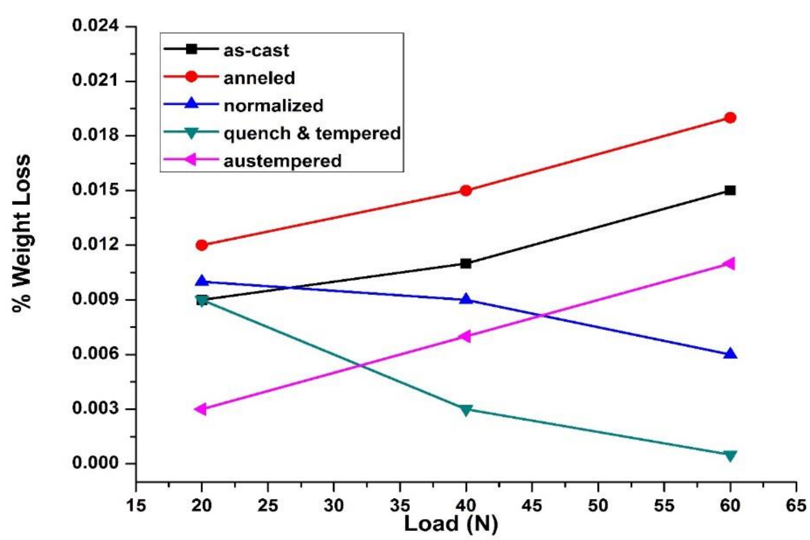


Fig 4.4- Diagram of weight loss Vs load for SG-01

From the figure 4.4, it is clear that for as cast bull's eye ferritic/ pearlitic matrix weight loss increases continuously when the load is increased from 20N to 60N. The reason behind this is the formation of ferritic ring around the graphite nodules which magnify the wear rate [22]. On the other hand, for annealed specimen weight loss increases continuously from 20N to 60N load, due to the presence of softer ferritic phase. In case of normalized specimen weight loss decreases with the increase in load from 20N to 60N. This is attributed to the fact of it is harder one. Furthermore, it is clear that for quenched and tempered specimen losses its weight continuously for every load between 20N to 60N. For the austempered specimen weight loss increases continuously from 20N to 60N load.

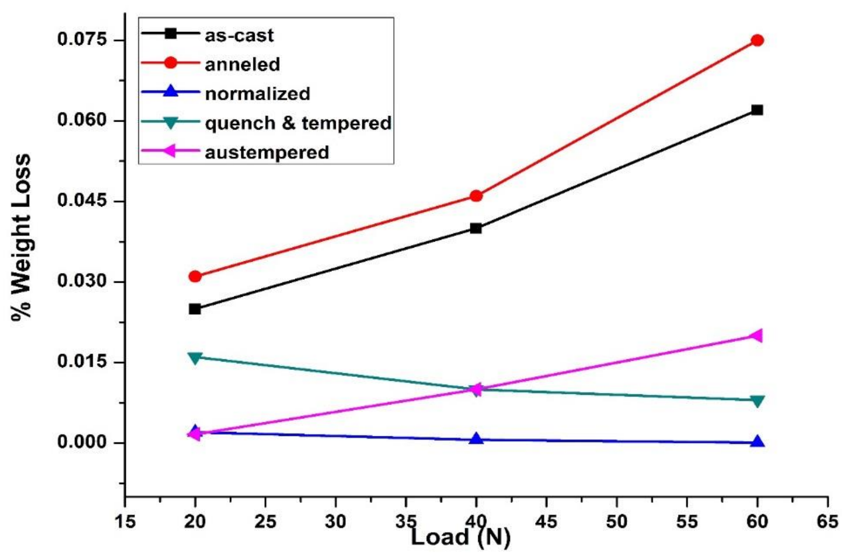
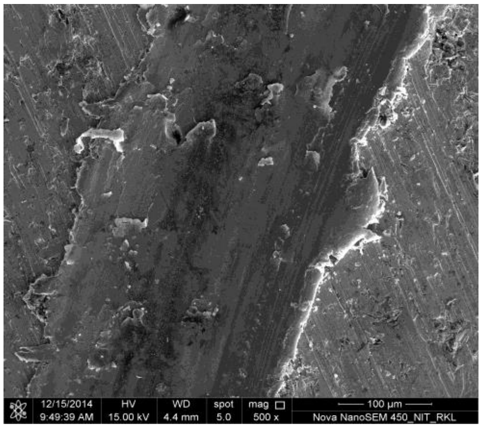
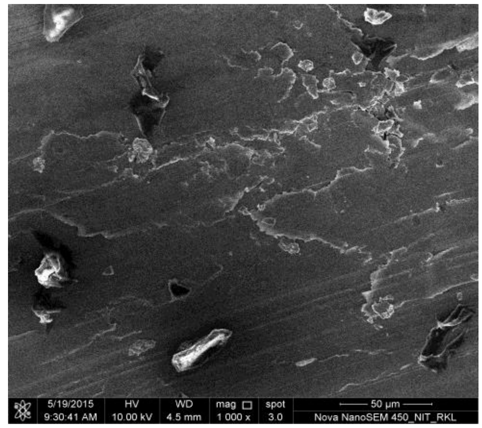


Fig 4.5- Diagram of weight loss Vs load for SG-02

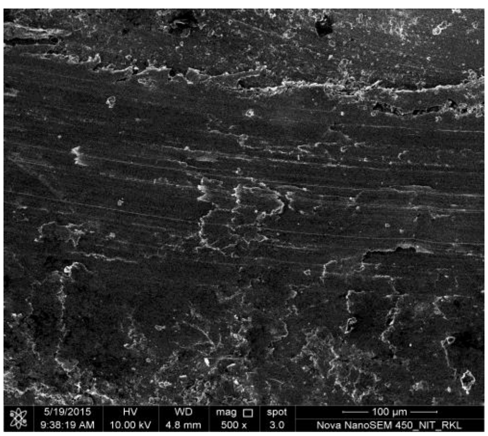
From the above figure, it is clear that for as-cast and annealed specimen weight loss increases when load increases from 20N to 60N. For normalized sample, the graph is almost straight in nature, there is no change in weight loss is observed. Furthermore, for quenched and tempered specimen weight loss decreases continuously from 20N to 60N load. For austempered specimen weight loss increases continuously up to load of 60N.

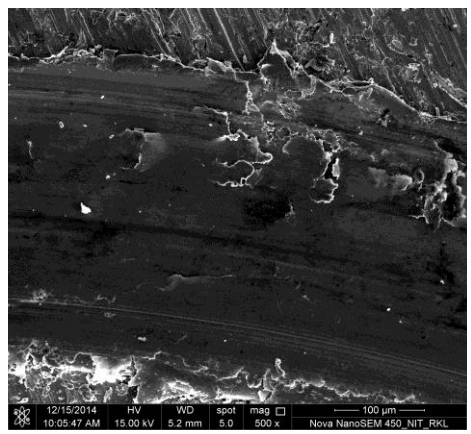
4.4 Wear mechanism- In order to determine the wear mechanism in different as-cast and heat treated specimen of SG-01 and SG-02 composition FESEM is conducted over the specimen.



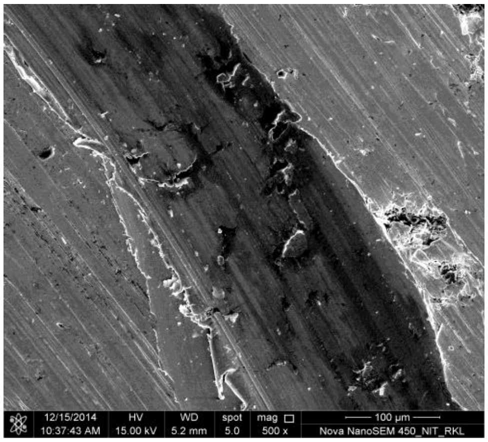


(1)As-cast specimen 20N (SG-01) **(2) As-cast specimen 20N (SG-02)**

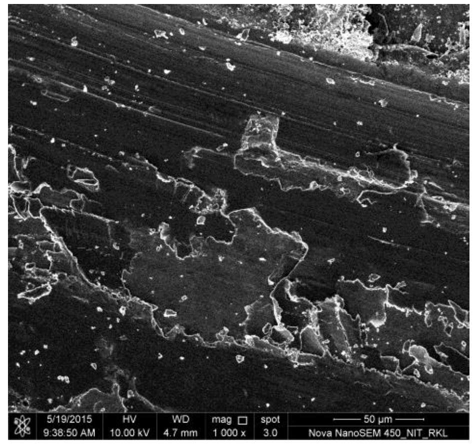




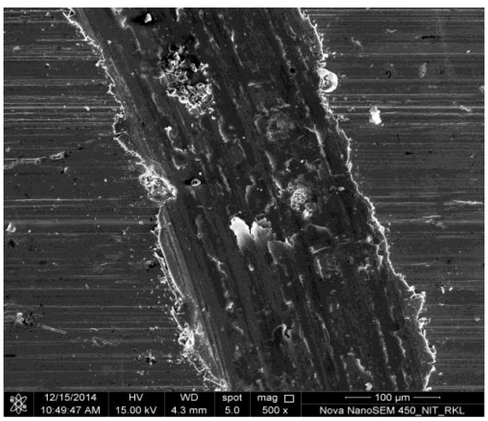
(3) Annealed specimen 20N (SG-01) **(4) Annealed specimen 20N (SG-02)**



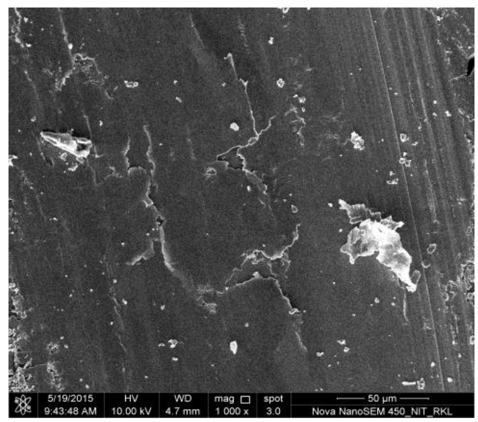
(5) Normalized specimen 20N (SG-01)



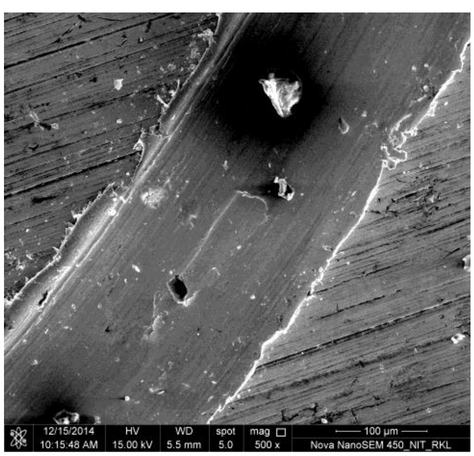
(6) Normalized specimen 20N (SG-02)



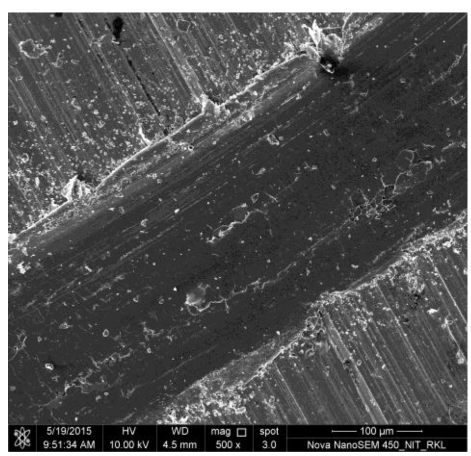
(7) Tempered specimen 20N (SG-01)



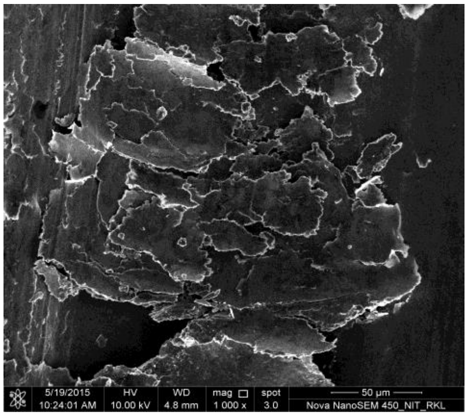
(8) Tempered specimen 20N (SG-02)



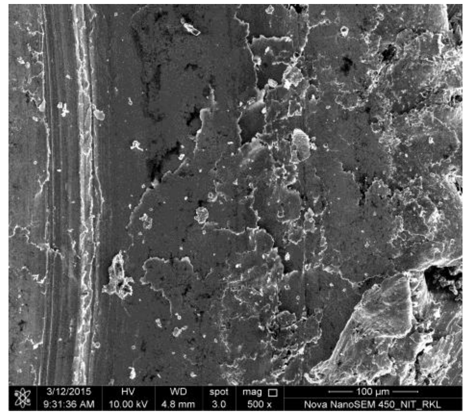
(9) Austempered specimen 20N (SG-01)



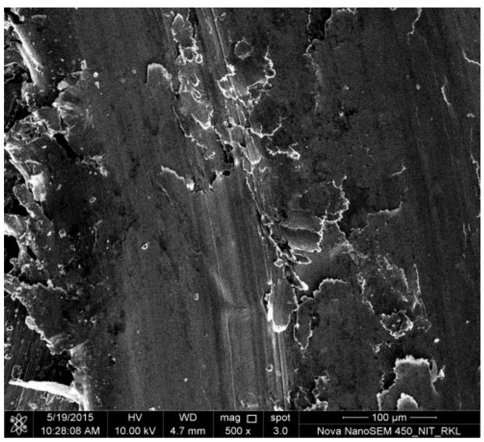
(10) Austempered specimen 20N (SG-02)



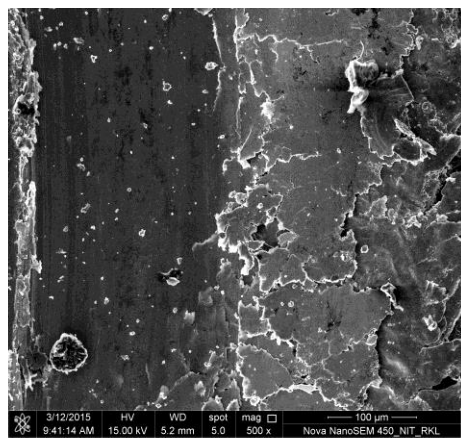
(11) As-cast specimen 40N (SG-01)



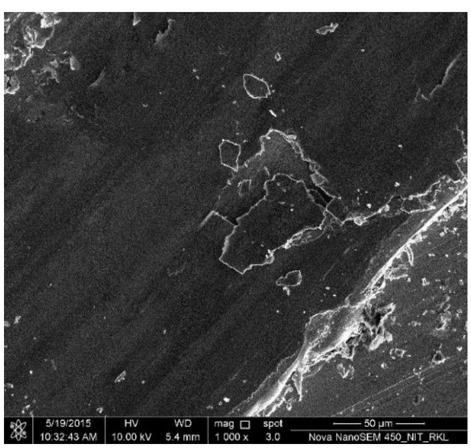
(12) As-cast specimen 40N (SG-02)



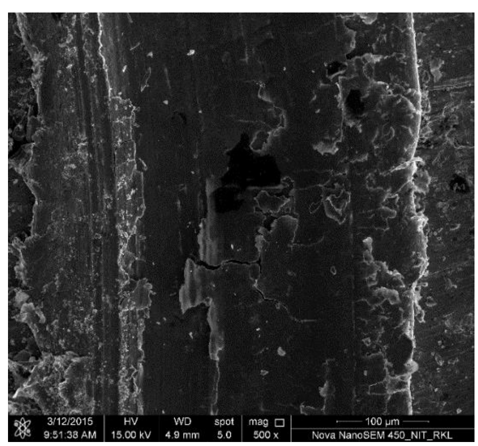
(13) Annealed specimen 40N (SG-01)



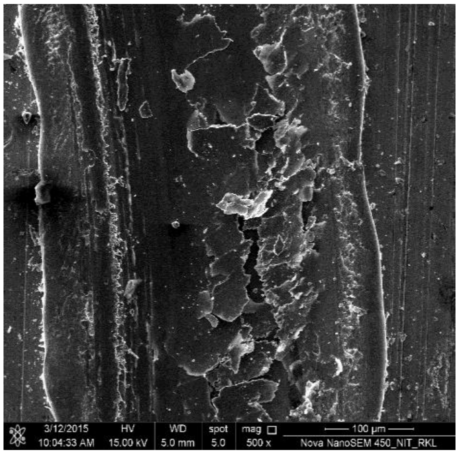
(14) Annealed specimen 40N (SG-02)

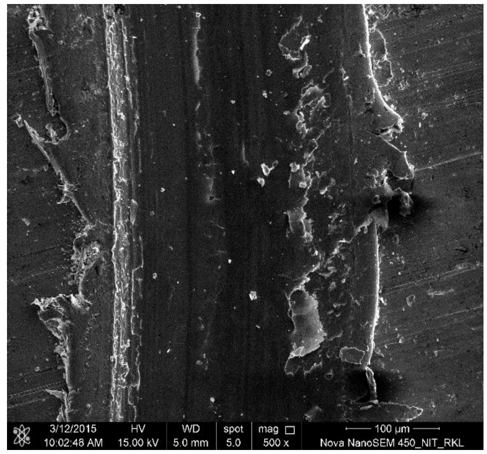


(15) Normalized specimen 40N (SG-01)

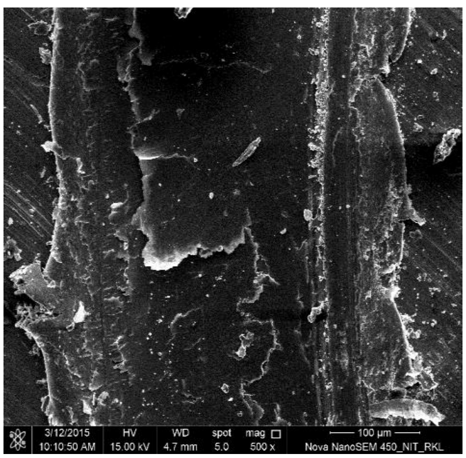


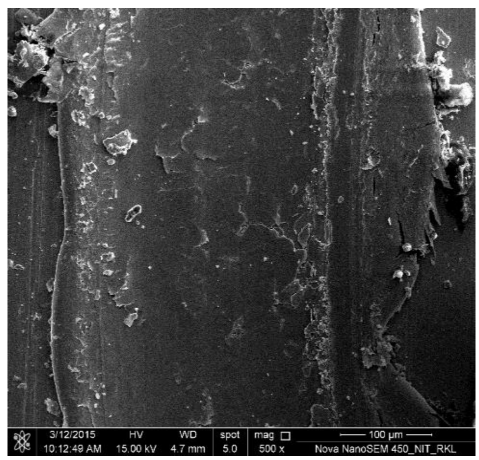
(16) Normalized specimen 40N (SG-02)



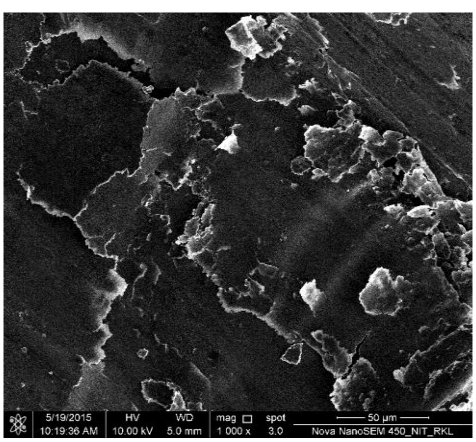


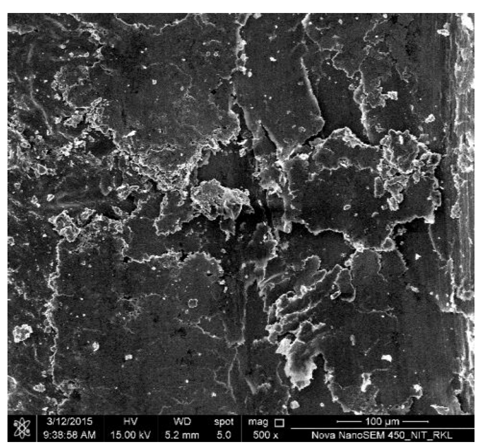
(17) Tempered specimen 40N (SG-01) **(18) Tempered specimen 40N (SG-02)**



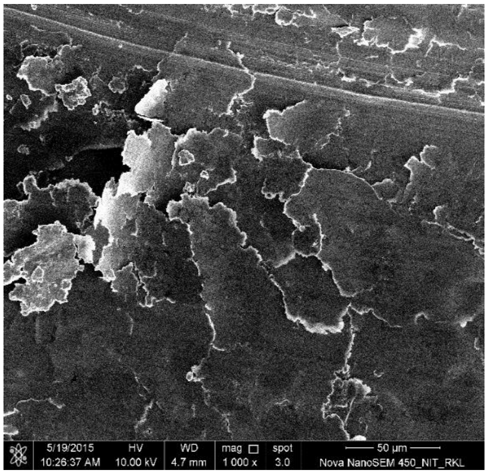


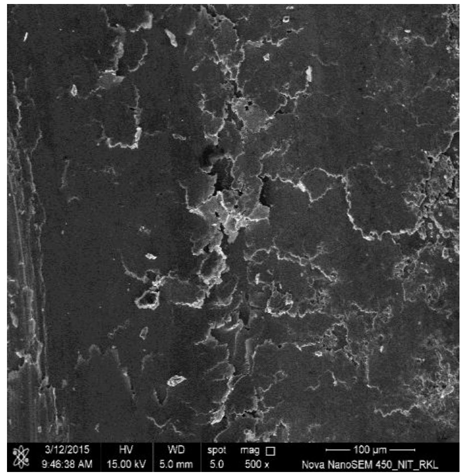
(19) Austempered specimen 40N (SG-01) **(20) Austempered specimen 40N (SG-02)**



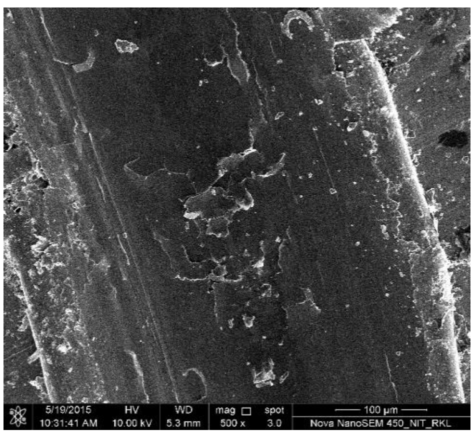


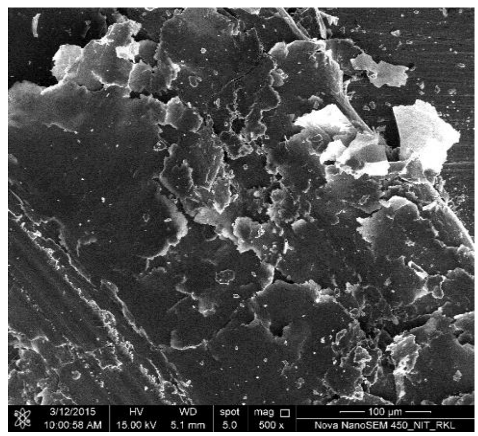
(21) As-cast specimen 60N (SG-01) **(22) As-cast specimen 40N (SG-02)**



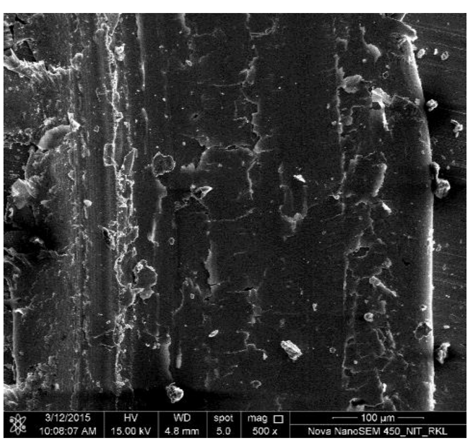


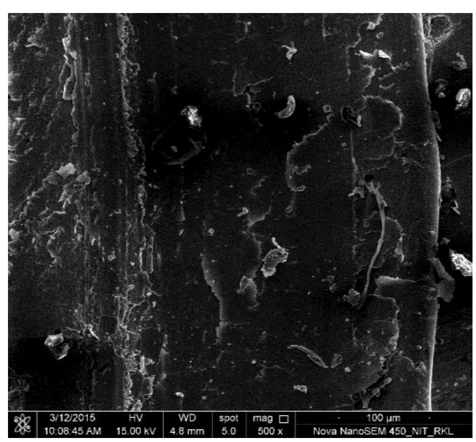
(23) Annealed specimen 60N (SG-01) **(24) Annealed specimen 60N (SG-02)**



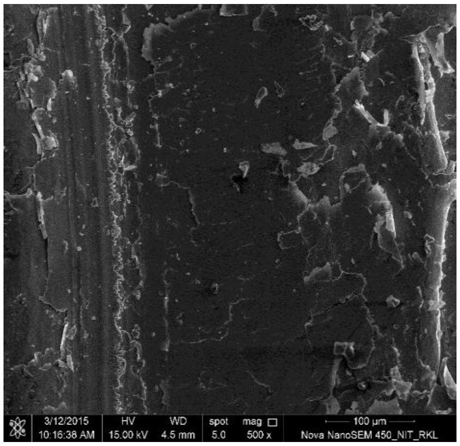


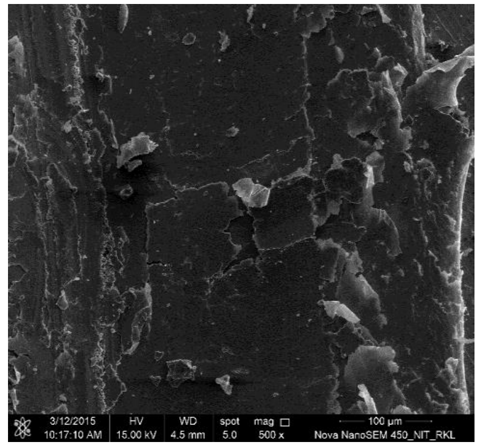
(25) Normalized specimen 60N (SG-01) **(26) Normalized specimen 60N (SG-02)**





(27) Tempered specimen 60N (SG-01) **(28) Tempered specimen 60N (SG-02)**





(29) Austempered specimen 60N (SG-01)   (30) Austempered specimen 60N (SG-02)

Fig 4.4.1: Images of Worn surface of As-cast and heat treated specimen of SG-01 and SG-02 by FESEM

From the investigation it was found that the wear mechanism involved at 20N load in as-cast and annealed specimens, is adhesive type of wear signified by the presence of delaminated layer without any cracks at the graphite/matrix interface or in the matrix phase. Furthermore, when specimen is subjected to the higher load plastic deformation begins to start at this graphite/matrix interface, since these are the sites of initiation of microcracks. Bull's eye structure of ferrite/pearlite in As-cast specimen of SG-01 composition may cause wear rate increases. This is attributed to the fact that ferritic phase around the graphite nodules is magnify the effect of wear behaviour. On the other hand the normalized, quench & tempered and austempered specimens with higher hardness values observed to have flat surfaces along with minor cracks on the worn surface at 20N load. But as the load increases specimen reached their plasticity stage and on continuous running of indentor over the surface plastic deformation occurred as can be observed by the cracks present on the worn surface.

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Chapter 5

Conclusion

5 Conclusions:

On the basis of the whole experiments, the following conclusions are drawn-

(1) The as-cast microstructure of SG-01 has bull's eye pearlitic/ferritic structure whereas that of SG-02 is fully ferritic, resulting higher hardness as the later one is the softest phase available in iron carbon binary system.

(2) On application of heat treatment processes the as-cast matrix transform to fully ferritic, pearlitic/ferritic, tempered martensitic and coarse upper bainitic for annealed, normalized, quench & tempered and austempered specimens respectively.

(3) The hardness value in both the cases are found to be for quenched and tempered specimen. The hardness value of every specimen for alloy SG-02 is higher than that of SG-01 and is attributed to the fact that presence of alloying elements like Mo, Cr, and higher amount of silicon that helps in solid solution strengthening in ferrite phase.

(4) The weight loss in case of softer materials is observed to be increased when load is increased continuously from 20N to 60N, while the hard phases appeared to have very good wear resistance capacity showing lower rate of weight loss with increase in load. The optimum condition in both alloys is found to be normalized specimen who showed almost no change in weight loss at every load.

(5) Wear mechanism involved in every case is adhesive type wear recognized by delaminated layer in softer matrices whereas plastically deformed cracks on worn surface of harder matrices are observed irrespective to the loading condition.

Chapter 6

Reference

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6 References:

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